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PATENT 1422-0401P



IN THE UNITED STATES PATENT & TRADEMARK OFFICE

In Re Application of: :  
Takayuki MITSUYA et al. : Group Art Unit: 1761  
Serial No.: 09/423,085 :  
Filed: November 2, 1999 : Examiner: MADSEN, Robert  
For: POWDER COMPOSITION

DECLARATION UNDER 37 C.F.R. 1.132

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WASHINGTON, D.C. 20231

Sir:

I, Noriyuki ISHIHARA, residing at Mie-ken, Japan, hereby declare and state as follows:

1. That I am familiar with the subject matter of U.S. Application No. 09/423,085.
2. That I received a bachelor's degree from Mie University, Department of Agriculture in the year 1987, majoring in agricultural chemistry, and that I received a master's degree from Mie University, Department of Agriculture in the year 1989, majoring in agricultural chemistry.

3. That I have been employed in Taiyo Kagaku Co., Ltd. in the year 1989 and have been assigned to the Research Laboratories.
4. That I have been involved in the research and development of functional foods.
5. That the following experiments were conducted by myself or under my direct supervision and control in order to verify that a comparative study of the powder composition of the present invention with the egg-white powder composition disclosed in U.S. Patent No. 5,127,953 (hereinafter simply referred to as "egg-white powder composition") reveals that the powder composition of the present invention is clearly different from the egg-white powder composition and exhibits unexpectedly excellent effects.

## METHODS

### (1) Preparation of Egg-White Powder Compositions

#### (1-1) Preparation of Egg-White Powder Composition Having Tea Extract and Soybean Oil

The egg-white powder composition having tea extract and soybean oil was prepared in accordance with the process of Example 6 of U.S. Patent No. 5,127,953 (hereinafter simply referred to as US'953), except that 23.4 parts by weight of tea extract (polyphenol content: 78% by weight, and caffeine content: 9% by weight) and 21.6 parts by weight of soybean oil were used in place of 45 parts by weight of corn oil, to give an egg-white powder composition having tea extract and soybean oil. Specifically, 23.4 parts by weight of tea extract (polyphenol content: 78% by weight,

and caffeine content: 9% by weight), 21.6 parts by weight of soybean oil and 55 parts by weight of egg-white powder having a water content of 6.7%, a packed bulk density of 0.54 g/ml and a spherical shape of 50 mesh pass (Tyler) were well kneaded manually by means of a stainless spatula for 1 min. in a 250 ml plastic vessel. Then 2.5 parts by weight of glycerin were added to the resultant mixture and then kneaded for 15 min. at 15°C, to give an egg-white powder composition having tea extract and soybean oil (hereinafter referred to as "Egg-White Powder Composition-A").

**(1-2) Preparation of Egg-White Powder Composition Having Tea Extract**

The egg-white powder composition having tea extract was prepared in accordance with the process of Example 6 of US'953, except that 23.4 parts by weight of tea extract (polyphenol content: 78% by weight, and caffeine content: 9% by weight) and 18.36 parts by weight of ethanol were used in place of 45 parts by weight of corn oil, to give an egg-white powder composition having tea extract. Specifically, 23.4 parts by weight of tea extract (polyphenol content: 78% by weight, and caffeine content: 9% by weight), 18.36 parts by weight of ethanol and 55 parts by weight of egg-white powder having a water content of 6.6%, a packed bulk density of 0.56 g/ml and a spherical shape of 50 mesh pass (Tyler) were well kneaded manually by means of a stainless spatula for 1 min. in a 250 ml plastic vessel. Then 2.5 parts by weight of glycerin were added to the resultant mixture and then kneaded for 15 min. at 15°C, to give an egg-white powder composition having tea extract (hereinafter referred to as "Egg-White Powder Composition-B").

(1-3) Preparation of Egg-White Powder Composition Having  $\beta$ -Carotene

The egg-white powder composition having  $\beta$ -carotene was prepared in accordance with the process of Example 6 of US'953, except that 2 parts by weight of  $\beta$ -carotene was used in place of 45 parts by weight of corn oil and that the amount of the egg-white powder was changed from 55 parts by weight to 98 parts by weight, to give an egg-white powder composition having  $\beta$ -carotene. Specifically, 2 parts by weight of  $\beta$ -carotene and 98 parts by weight of egg-white powder having a water content of 6.5%, a packed bulk density of 0.55 g/ml and a spherical shape of 50 mesh pass (Tyler) were well kneaded manually by means of a stainless spatula for 1 min. in a 250 ml plastic vessel. Then 2.5 parts by weight of glycerin were added to the resultant mixture and then kneaded for 15 min. at 15°C, to give an egg-white powder composition having  $\beta$ -carotene (hereinafter referred to as "Egg-White Powder Composition-C").

(1-4) Preparation of Egg-White Powder Composition Having Fish Oil

The egg-white powder composition having fish oil was prepared in accordance with the process of Example 6 of US'953, except that 3 parts by weight of fish oil (DHA content: 25% by weight) was used in place of 45 parts by weight of corn oil and that the amount of the egg-white powder was changed from 55 parts by weight to 97 parts by weight, to give an egg-white powder composition having fish oil. Specifically, 3 parts by weight of fish oil (DHA content: 25% by weight) and 97 parts by weight of egg-white powder having a water content of 6.7%, a packed bulk density

of 0.54 g/ml and a spherical shape of 50 mesh pass (Tyler) were well kneaded manually by means of a stainless spatula for 1 min. in a 250 ml plastic vessel. Then 2.5 parts by weight of glycerin were added to the resultant mixture and then kneaded for 15 min. at 15°C, to give an egg-white powder composition having fish oil (hereinafter referred to as “Egg-White Powder Composition-D”).

(2) Preparation of Powder Compositions

(2-1) Preparation of Powder Composition Impregnated with Tea Extract and Soybean Oil

In accordance with the method described in Example 1 of the present specification, a powder composition impregnated with tea extract and soybean oil was prepared. Specifically, in 8 kg of a soybean oil was homogeneously dispersed 5.5 kg of a tea extract (polyphenol content: 78% by weight, caffeine content: 9% by weight), and the resulting dispersion was added to 10 kg of delipidated egg yolk particles. The mixture was stirred at 30°C for 50 minutes with a vacuum kneader (manufactured by Kajiwara Kogyo K.K. under the trade name of KDV-5E; 30 mmHg), so that the dispersion was dispersed and impregnated therein, to give 23.5 kg of a powder composition (average particle size: 40  $\mu$ m, water content: 4.2% by weight) impregnated with tea extract and soybean oil (hereinafter referred to as “Powder Composition-A”).

The delipidated egg yolk particles used in this example were prepared in accordance with Preparation Example 1 of the present specification. Specifically, to 100 kg of egg yolk powder was added 2000 liter of ethanol, and the mixture was

stirred with a homomixer at 40°C for 30 minutes. The resulting mixture was filtered with a flat plate-type filtration apparatus using a filter paper. To the resulting filtration residue was added 200 kg of deionized water, and mixed. Thereafter, the mixture was dried with a spray-dryer (manufactured by Ohgawara Kakoki under the trade name of Model "DC16", inlet: 140°C, outlet: 75°C), whereby giving 32 kg of delipidated egg yolk particles which were porous, having a large number of pores on the particle surface. The pores were confirmed with SEM. The pore size was such that the diameter was about 0.1 to about 10  $\mu\text{m}$ .

#### (2-2) Preparation of Powder Composition Impregnated with Tea Extract

In accordance with the method described in Example 2 of the present specification, a powder composition impregnated with tea extract was prepared. Specifically, in 12 kg of ethanol was homogeneously dispersed 8 kg of a tea extract (polyphenol content: 78% by weight, caffeine content: 9% by weight), and the resulting dissolved dispersion was added to 10 kg of the delipidated egg yolk particles obtained in the same manner as in item (2-1). The mixture was stirred at 30°C for 50 minutes with a vacuum kneader (30 mmHg) (manufactured by Kajiwara Kogyo K.K. under the trade name of KDV-5E; 30 mmHg), so that the dispersion was dispersed and impregnated therein, to give 18 kg of a powder composition (average particle size: 40  $\mu\text{m}$ , water content: 3.4% by weight) impregnated with tea extract (hereinafter referred to as "Powder Composition-B").

#### (2-3) Preparation of Powder Composition Impregnated with $\beta$ -Carotene

In accordance with the method described in Example 3 of the present specification, a powder composition impregnated with  $\beta$ -carotene was prepared. Specifically, to 10 kg of delipidated egg yolk particles was added 2 kg of a 30%  $\beta$ -carotene containing plant oil suspension having a distinctive odor. The mixture was stirred at 30°C for 30 minutes with a vacuum kneader (30 mmHg) (manufactured by Kajiwara Kogyo K.K. under the trade name of KDV-5E; 30 mmHg), so that the dispersion was dispersed and impregnated therein, to give 12 kg of a powder composition (average particle size: 34  $\mu$ m, water content: 5.1% by weight) impregnated with  $\beta$ -carotene (hereinafter referred to as "Powder Composition-C").

The delipidated egg yolk particles used in this example were prepared in accordance with Preparation Example 2 of the present specification. Specifically, to 100 kg of egg yolk powder was added 2000 liter of ethanol, and the mixture was stirred with a homomixer at 40°C for 30 minutes. The resulting mixture was filtered with a flat plate-type filtration apparatus using a filter paper. To the resulting filtration residue was added 100 kg of deionized water, and mixed. Thereafter, the mixture was dried with the spray-dryer (manufactured by Ohgawara Kakoki under the trade name of Model "DC16", inlet: 140°C, outlet: 75°C), whereby giving 32 kg of delipidated egg yolk particles which were porous, having a large number of pores (size: about 0.1 to about 10  $\mu$ m) on the particle surface as confirmed by SEM.

#### (2-4) Preparation of Powder Composition Impregnated with Fish Oil

In accordance with the method described in Example 6 of the present specification, a powder composition impregnated with fish oil was prepared.

Specifically, to 10 kg of the delipidated egg yolk particles obtained in the same manner as in item (2-1) was added 3 kg of a fish oil (DHA content: 25% by weight). The mixture was stirred at 30°C for 1 hour with a vacuum kneader (30 mmHg) (manufactured by Kajiwara Kogyo K.K. under the trade name of KDV-5E; 30 mmHg), to give 12.8 kg of a powder composition (average particle size: 46 µm, water content: 5.6% by weight) impregnated with fish oil (hereinafter referred to as "Powder Composition-D").

## TESTS AND RESULTS

### (1) Evaluation of Egg-White Powder Compositions-A and -B, and Powder Compositions-A and -B

Sensory examination of Egg-White Powder Compositions-A and -B, and Powder Compositions-A and -B was carried out in accordance with the method described in Test Example 2 of the present specification. Specifically, panelists consisting of 5 each of male and female normal individuals of an age of 24 to 34 years old were subjected to sensory examination for "bitterness" by placing 20 mg of each powder composition on the tongue. The evaluation criteria are as follows.

#### Evaluation Scores

0	No bitterness;
1	Slight bitterness;
2	Little bitterness;
3	Some bitterness;

- 4            Marked bitterness; and  
 5            Strong bitterness.

The value obtained by dividing the total sum of the evaluation scores for "bitterness" for each individual panelists by the total number of the panelists (10 persons) was defined as sensory evaluation for "bitterness".

The results are as follows.

<u>Sample</u>	<u>Evaluation Score for Bitterness</u>
Egg-White Powder Composition-A	4.8
Egg-White Powder Composition-B	4.8
Powder Composition-A	1.2
Powder Composition-B	1.8

It can be seen from the above results that the powder compositions of the present invention favorably suppressed the exhibition of undesired bitterness of the tea extract, while the egg-white powder compositions prepared in accordance with the method of US'953 could not suppress the exhibition of undesired bitterness of tea extract.

(2) Evaluation of Egg-White Powder Composition-C and Powder Composition-C

The POV measurement and the sensory examination were carried out for Egg-White Powder Composition-C and Powder Composition-C in accordance with the

method described in Test Example 3 of the present specification. Specifically, each powder composition in an open system was respectively stored at 60°C in dark place. The POV measurement and the sensory examination were carried out with the passage of time.

As a result, the POV measured for Egg-White Powder Composition-C was stable until 10 days after, but reached the level of 50 meq/kg after 30 days. On the other hand, the POV measurement for Powder Composition-C was 0.5 meq/kg, remarkably stable even after 30 days.

(3) Evaluation of Egg-White Powder-D and Powder Composition-D

POV measurement and sensory examination were carried out for Egg-White Powder Composition-D and Powder Composition-D in the same manner as in item (2).

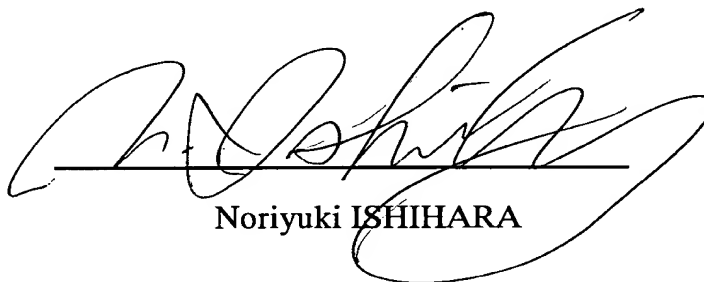
As a result, the obtained value for Egg-White Powder Composition-D was stable until 5 days after, but reached the level of 60 meq/kg after 30 days with generation of unpleasant odor. On the other hand, the obtained value for Powder Composition-D was 0.5 meq/kg, remarkably stable even after 30 days, and generation of unpleasant odor was not also detected.

## DISCUSSION

It is clear from above that the powder composition of the present invention is completely different from the egg-white powder composition disclosed in US'953. This means that the delipidated egg yolk powder used in the present invention is completely different from the egg-white powder composition of US'953. The test results show that the effects of the present invention regarding prevention of the deterioration of a substance and improvement of flavor cannot be obtained when the egg-white powder composition of US'953 is impregnated with substances that have undesirable flavor, or substances that are susceptible to deterioration as used in the present invention. Therefore, the superior effects exhibited by the powder composition of the present invention are never expected from the egg-white powder composition of US'953.

6. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

7. Further declarant saith not.



Noriyuki ISHIIHARA

October 29, 2002

Date